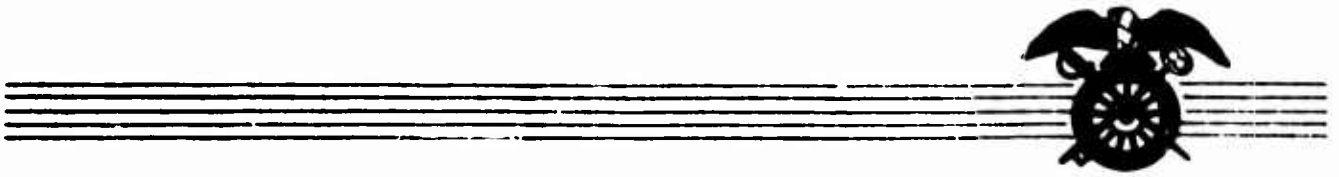
A-338 - Rpt #8(Final)
Contract: DA19-129-qm-1540
NRC Equipment Corporation

Improving Freeze Drying Process Efficiency through Improved Vapor Removal and In-Process Moisture Determination

Period: 15 January 1960 - 30 June 1962



ARMED FORCES FOOD AND CONTAINER INSTITUTE
U. S. Army Research and Engineering Command
Chicago 9, Illinois

AD _____ Accession No. _____ UNCLASSIFIED

NRC Equipment Corp., Newton 61, Mass.

1. Dehydrated Meat
2. Contract DA-19-129-QM-1546

IMPROVING FREEZE-DRYING. PROCESS EFFICIENCY THROUGH IMPROVED VAPOR REMOVAL AND IN-PROCESS MOISTURE DETERMINATION

Dr. B. Kan
Report No. 8, July 31, 1962, (Contract - DA-19-129-QM-1546)

Unclassified Report
42 pages
Proj. 7-84-06-032

Many ways of following the course of freeze drying processes and of controlling their progress have been examined. Of these, the methods utilizing measurements of chamber atmosphere, surface temperature measurement and moisture analysis by measurement of RF power absorption appear most promising and all merit further examination for application on full scale drying units.

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CONTRACT RESEARCH PROJECT REPORT

QUARTERMASTER FOOD AND CONTAINER INSTITUTE FOR THE ARMED FORCES, CHICAGO
Research and Engineering Command, U. S. Army, QM Research and Engineering Center, Natick, Massachusetts.

NRC Equipment Corporation
160 Charlemont Street
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Official Investigator -
Dr. B. Kan

Project No. 7-84-06-032
Contract No. DA-19-129-QM-1546
File No. A-338
Report No. 8 (Final)
Period 15 January, 1960
to June 1962

Collaborators -
R. A. Yeaton
R. E. Elwinger
Title of the Contract - Improving Freeze-Drying Process Efficiency Through Improved Vapor Removal and In-Process Moisture Determination.

Introduction

Freeze dehydration is a process for drying food from the frozen state. This is generally achieved by placing frozen food in a vacuum chamber and then reducing the system pressure below the triple point of the phase diagram for water (roughly 4.6 torr). Under these conditions, the ice in the frozen food can be sublimed by the application of heat and the food solids remain as a mass of porous material without undergoing the shrinkage that is characteristic of food whose water has been removed by evaporation from the liquid phase. This lack of shrinkage together with the fact that the dehydration is carried out at low temperature and in the absence of oxygen generally means that freeze dehydrated products are markedly superior to air dried foods in quality and therefore are of considerable current interest to both the food industry and the Armed Forces of the United States.

At the present time the process is lengthy and the cost of removing water by this means is usually calculated to be higher than for other dehydration methods. It was the original aim of this project to study the processing variables in order to arrive at optimum conditions for short drying cycles and also to find a method of moisture analysis that could be used to follow the progress of the dehydration so that the cycle could be stopped immediately after the proper moisture content was achieved.

Initial studies on the effects of radiator temperature and chamber pressure on the length of the drying cycle appeared to show that there was a direct relationship between radiator temperature and drying rate; however, no clear effect of chamber pressure on drying rate could be discerned. It was found when drying by radiant heat transfer (with the food supported out of contact with the surface of the lower radiator) that initial radiator temperatures could be higher than 400°F for a brief period of time at the beginning of the drying cycle if they were subsequently lowered for the rest of the cycle. Obtaining a comparison of the effects of the various conditions on "cycle time" proved difficult because it was not possible to determine the end of the process in a precise manner. Accordingly the project was directed to finding improved methods of end point determination.

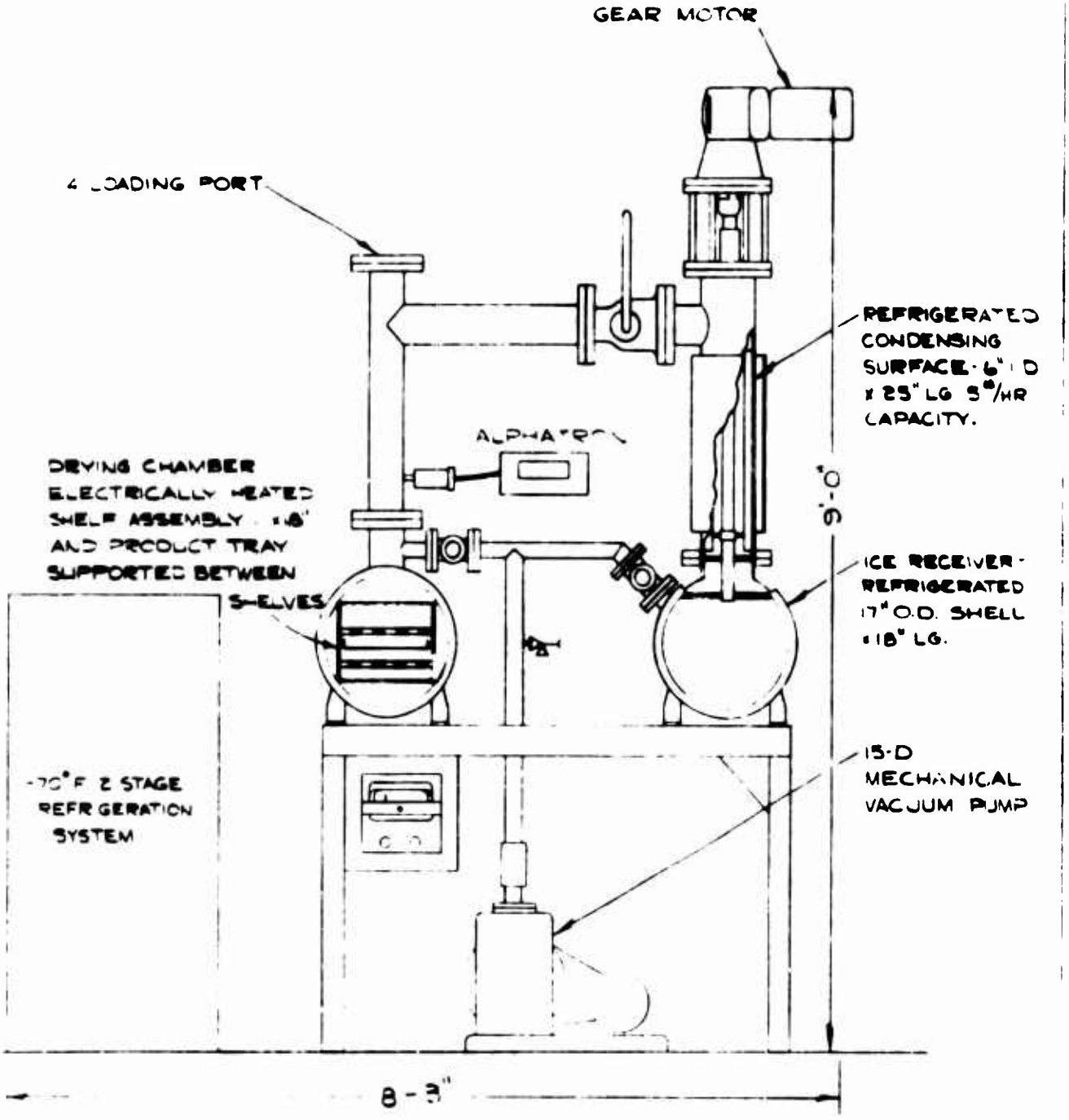
This final report is primarily concerned with the investigation of end point determination. The Appendix includes summaries of the contents of the progress reports which include details of other project work.

Due to the independent nature of the various phases of this project this report will not be divided into "procedures, results and discussion" sections on an overall basis, but will take the form of a series of independent reports on the various studies. An immediate exception will be the first section covering procedures used throughout the study and a description of the freeze drying equipment. Subsequently the separate studies will be presented roughly in chronological order but with emphasis on logical development of thoughts.

Procedures and Equipment

A. Freeze drying unit

Figure 1 is an outline drawing of the freeze drying unit used in these studies. It consisted of a cylindrical drying chamber heated by electrical strip heaters fixed around its outside wall. Inside the drying chamber was a stainless steel paddle wheel that mixed the contents during drying. This set-up was used in an initial series of experiments. Subsequently, the paddle wheel was removed and a stainless steel mesh was placed in the chamber and food placed on this mesh was dried by thermal radiation from the hot wall. Finally a set of two shelves was built. Each shelf consisted of four Chromalox electrical



OUTLINE

FREEZE DRYING PILOT PLANT

NRC EQUIPMENT CORP

FIG 1

strip heaters sandwiched between two 1/8" thick blackened aluminum plates. The shelves were supported in a rack so that the distance between the plates was adjustable. Both heating systems were electrically controlled and could reach 400°F in temperature.

The vapor handling system was not changed during this project. It consisted of a NRC V-5D rotary scraped condenser and refrigerated ice receiver, both cooled by a two stage refrigeration system with a capacity of condensing five pounds of ice an hour at -68°F.

A 15 CFM rotary mechanical pump was used for initial evacuation of the chamber and for handling non-condensable vapors during drying runs.

A four inch ball valve was located so that it could be used to separate the chamber from the vapor handling system.

B. Moisture Analysis

Moisture was determined gravimetrically after drying overnight in a vacuum oven at 100°C as prescribed in the Official Methods of Analysis of the Association of Official and Agricultural Chemists (Eighth Edition, 1955, method 23.2).

In the work with the dielectric moisture analyzer the large mass of water that often had to be removed did not permit use of the drying oven. It was therefore necessary to dry these samples overnight in the freeze dryer at somewhat lower temperatures.

Initial Studies

The first runs were made in the NRC rotary freeze dryer. Approximately 20 pounds of 1/2" x 1/2" x 1" beef sticks were loaded into the unit and dried under high vacuum while being mixed by a rotating paddle wheel and heated by the chamber wall. Chamber pressure was controlled by air or nitrogen bleed. In order to obtain uniform particle size the beef sticks were prepared by sawing frozen beef to the desired dimensions.

In these runs an attempt was made to follow the progress of the dehydration by taking periodic samples of beef from the dryer for moisture analysis. This procedure was found not to be useful because the drying of the individual sticks was not uniform and it was not pos-

sible to withdraw a representative sample. This procedure was not investigated further.

Alphatron(R) - McLeod Gauge Comparison

In the above runs the differences in values of McLeod and Alphatron vacuum gauges were taken and were examined for their possible usefulness as a control reading.+ The procedure was based on the belief that McLeod gauges measure only the partial pressure of non-condensable vapors and the Alphatron(R) measures the sum of the partial pressures of all vapors present.

The results of four of these runs are shown in figures 2 through 5. There is no apparent relationship between the differences in gauge readings and progress of the drying cycle. In figures 4 and 5 there is some tendency for the McLeod pressure gauge readings to rise towards the end of the drying cycle. This phenomenon is probably related to the one observed and reported below in the section on the Vapor Sample Condensing System. At the time this work was done full attention was devoted only to the comparison of the two gauge readings and, since these were not fruitful, the comparative readings were abandoned.

The rise in McLeod gauge reading was not further investigated. In retrospect, it appears possible that controlling the pressure by bleeding in non-condensable gas was partly responsible for the inconsistent results obtained.

(R) Reg. US Pat. Off.

+ These gauges are described in "Scientific Foundations of Vacuum Technique" - S. Dushman and J. M. Lafferty, John Wiley and Sons, New York, 1962.

Figure 2

DATA

Run 1 Beef, top round, good

Time	Alphatron® Pressure Microns	McLeod Pressure Microns	Difference Microns	
8:30 AM	1200	Off Scale	-	Start
9:00	300	180	120	
9:30	240	150	90	
10:00	200	110	90	
10:30	190	110	80	
11:00	700	150	550	
11:30	660	400	260	
1:00 PM	760	180	580	
1:30	700	170	530	
2:00	540	150	390	
2:30	500	-	-	
3:00	500	120	380	
3:30	560	120	460	
4:00	580	105	475	

Held in freezer overnight - 14 lbs. 5 oz. ice removed.
Run continued next morning.

8:00 AM	760	Off Scale	-	
8:30	760	75	685	
9:00	620	170	450	
9:30	560	190	370	
10.00	760	255	505	
10:30	760	280	480	
11:00	760	270	490	
11:30	720	85	635	
1:00	570	140	430	
1:30	800	150	650	
2:00	820	160	660	
2:30	620	240	380	
3:00	640	360	280	Heat off 3:15
3:30	760	330	430	

	Weight	Moisture	Fat
Initial	23 lbs. 6 oz.	72.2%	12 %
Final	3 lbs. 10 oz.	1.4%	32.4%

Rehydration -131%
Chamber Wall Temperature -150°F

Figure 3

Run 2 Beef, top round, commercial

Time	Alphatron® Pressure Microns	McLeod Pressure Microns	Difference Microns
8:00 AM	700	210	490
8:30	520	70	450
9:00	580	60	520
9:30	480	60	420
10:00	820	200	620
10:30	900	70	830
11:00	710	220	490
11:30	720	100	620
1:00 PM	660	75	585
1:30	720	100	620
2:00	700	75	625
2:30	1000	150	850
3:00	760	80	680
3:30	900	85	815
4:00	960	120	840

	Weight	Moisture	Fat
Initial	18 lbs. 14 oz.	70.9 %	25 %
Final	5 lbs. 9 oz.	1.25%	19.9%

Rehydration -208%
Chamber Wall Temperature -200°F

Figure 4

Run 3 Beef, top round, utility

Time	Alphatron® Pressure Microns	McLeod Pressure Microns	Difference Microns	"Product Temperature" °F
8:00 AM	800	700	100	22
8:30	800	60	740	32
9:00	720	40	680	36
9:30	910	60	850	41
10:00	850	50	800	45
10:30	760	50	710	45
11:00	740	50	690	47
11:30	680	50	630	48
1:00 PM	760	60	700	53
1:30	700	65	635	55
2:00	680	60	620	56
2:30	880	75	805	58
3:00	860	65	795	60
3:30	720	65	655	63
4:00	720	65	655	65
Held frozen overnight - 10 lbs. 8 oz. ice removed				
8:30 AM	700	340	360	-
9:00	1000	280	720	49
9:30	740	40	700	59
10:00	520	45	475	67
10:30	660	50	610	74
11:00	830	80	750	81
11:30	740	60	680	90
1:00 PM	620	150	470	100
1:30	560	400	160	100

	Weight	Moisture	Fat
Initial	7 lbs. 15 oz.	73 %	13.3%
Final	4 lbs. 13 oz.	1.1%	12.0%

Rehydration -253%
Chamber Temperature -200°F

Figure 5

Run 4 Beef, top round, choice

Time	Alphatron® Pressure Microns	McLeod Pressure Microns	Difference Microns	Chamber Wall Temp. °F	Product Temp. °F
8:00	660	340	320	230	30
8:30	660	60	600	250	33
9:00	520	60	460	250	33
9:30	640	70	570	250	44
10:00	800	75	725	250	46
10:30	700	75	625	250	50
11:00	700	75	625	250	52
11:30	680	75	605	250	53
1:00	710	70	640	250	58
1:30	740	100	640	200	60
2:00	820	75	745	200	59
2:30	780	85	695	200	59
3:00	740	75	665	200	62
3:30	920	80	840	200	65
4:00	800	60	740	200	68
	Product held frozen overnight.				
8:00	800	275	525	160	cold
8:30	780	85	695	200	51
9:00	670	45	625	200	66
9:30	540	40	500	200	78
10:00	740	50	690	200	96
10:30	820	50	770	170	100
11:00	840	120	720	150	100

	Weight	Moisture	Fat
Initial	23 lbs. 13 oz.	74.5%	10.9%
Final	6 lbs. 9 oz.	1.9%	25.1%

Rehydration - 231%

Moisture Distribution Within Charge During Freeze-Drying

It has been observed in earlier work that, if a freeze drying run has been stopped short of completion, residual ice may occur in a few isolated pieces and that the amount of residual ice can be quite variable. This variation can be attributed to non-uniformity of heat supply due to uneven radiator temperatures or differing amounts of shadowing by adjacent pieces. These variations in drying rate are of importance because at present the length of a drying cycle is determined by the slowest drying piece. In addition, a non-uniform load tends to complicate the task of determining cycle end points since acceptably low overall moisture content values will not insure that all individual pieces are ice-free. It was therefore decided to perform some experiments to elucidate the degree of non-uniformity that is being encountered in the drying of beef sticks.

A series of runs was made in the freeze dryer at each of two temperatures: 150°F and 200°F. Thirty beef sticks were placed in the central zone of an aluminum mesh tray in a rectangular array and spaced approximately one half inch apart. This was in order to minimize the effect of non-uniform heat supply. The product was dried while supported between the electrically heated plates for varying periods of time and was then removed and alternate pieces were analyzed for moisture.

Fifteen randomly selected beef sticks were analyzed for initial moisture content. The mean moisture content was 74.7% and the range was 72.7% to 76.3%.

The results of the drying runs are shown in the following table. They are expressed as percent moisture content and are shown in the same relative positions they occupied during drying. There is apparently no tendency for sticks in any single position to dry faster than in any other position suggesting that variations in drying rate due to variations in heat input were successfully minimized.

Moisture Contents of Freeze-dried Beef Sticks Arranged According to their Positions During Drying:

3 hours at 200°F

33.9%	27.4%	17.3%		
26.9%	30.5%	12.3%		
10.9%	10.3%	14.5%		
16.7%	21.1%	2.2%		
7.0%	12.2%	25.8%		all had wet centers

4 nours at 200°F

*3.7%	1.4%	1.7%		
*4.3%	0.9%	1.7%		
1.5%	2.5%	1.7%		
1.8%	1.1%	1.3%		
*6.8%	3.0%	1.2%		*wet centers

5 hours at 200°F

0.0%	0.0%	0.0%		
0.6%	0.4%	0.3%		
0.6%	0.6%	0.6%		
0.8%	0.4%	0.8%		
1.3%	0.9%	0.9%		all were dry

4 hours at 150°F

15.8%	28.6%	6.3%		
10.0%	25.0%	19.2%		
30.9%	16.2%	26.9%		
21.9%	23.7%	5.7%		
21.5%	27.6%	2.8%		all had wet centers

5 hours at 150°F

1.7%	*2.9%	1.9%	
1.9%	*14.5%	1.8%	
1.8%	*10.0%	1.7%	
*15.4%	2.1%	1.4%	
1.5%	1.5%	1.6%	*wet centers

6 hours at 150°F

0.9%	1.1%	*4.5%	
1.1%	1.5%	0.8%	
1.0%	1.4%	1.7%	
1.6%	1.7%	1.5%	
1.2%	1.2%	1.5%	*wet center

These results demonstrate that in any load of food being dried considerable variation of individual moisture contents can exist. At the end of the drying cycle these small masses of moisture require considerably more time to remove than an equal mass at the beginning of the drying cycle. These experiments suggest that towards the end of a drying cycle a very few pieces of food containing ice cores requires the drying process to be extended beyond the point where the major part of the load is satisfactorily dry. One can therefore conclude that the average moisture content of the load must be reduced to a value lower than the specified maximum in order to be assured that all individual pieces are at a satisfactory moisture level.

Mass Transfer Measurement

An obvious approach to the control of freeze dehydration cycles is the monitoring of mass transfer of water vapor. If the initial moisture content of the charge were known, and the weight loss of the charge were measured, it should be possible to determine the average moisture of the charge at all points of the cycle. It is possible to conceive of various related measurements that would achieve similar results such as weighing the ice removed or integrating vapor flow rates.

On an experimental scale the measurement of weight loss is certainly of value in assessing the effects of processing variables. On a commercial scale it is necessary to know the rate of vapor evolution as a function of time in order to design equipment properly. As an end point indicator, however, the measurement of weight loss is not an attractive procedure. It is in the first place difficult to obtain an accurate value of the starting moisture content and, secondly, the asymptotic drying curve results in very small changes in mass per unit of time at the end of the cycle.

It is not necessary at this point to completely reject all mass related methods of end point sensing, since it may be possible to make use of an in-process moisture analysis to obtain readings in various parts of a freeze dryer. This procedure would enable one to sense the presence of wet spots without having to consider this localized moisture as part of the moisture content of the entire load. This subject will be covered in greater detail below.

Thermocouples as End-Point Indicators

It is a common practice today in commercial freeze dehydration plants to imbed thermocouples in the food being freeze-dried in order to follow the progress of the drying cycle. Towards the end of a drying cycle the evaporative cooling due to sublimation of the ice is reduced and the internal temperature of the product rises. The process end point is taken to be that time when product temperature reaches some predetermined point.

Thermocouple measurements under these conditions are subject to error for a number of reasons. The manner in which the couple is imbedded will influence the reading. The exact location of the couple in the piece of food will also influence the reading. It is not only likely that heat transfer down the thermocouple will affect the temperature in the immediate vicinity of the junction, but it may even increase drying rate of the test piece. But aside from inaccuracy the method suffers from a sampling problem, and there is no assurance that the test pieces are representative of the slowest drying pieces in the load.

Consequently, it is usually considered prudent to dry the load for some additional period of time beyond the indicated end-point to insure that no ice remains in any other part of the load.

In spite of these limitations this technique is commonly used to monitor freeze drying processes simply because no better method is presently available.

Vapor Pressure Rise

A technique that has long been used in vacuum process work is the measurement of the change in pressure that occurs when the chamber is isolated from the pumping system. One such application has been in the control of metal degassing processes.

The taking of the vapor pressure rise measurement (henceforth referred to as VPR) involves closing the four inch ball valve and observing the change in chamber pressure on an Alphatron(R) vacuum gauge. It is possible to choose any arbitrary time period over which to measure the VPR, but in these experiments it was found convenient and effective to measure the VPR over a period of ten seconds.

Obviously the pressure must be allowed to rise enough to produce a readable change in the pressure gauge, but should never go so high as to result in thawing of the product.

An attempt was made at the outset to correlate the VPR with ice temperature. It was confirmed that both the temperature of ice cubes and of ice in a beef stick could be controlled by controlling the partial pressure of water vapor in the drying chamber, but no relationship between ice temperature and VPR was found. Instead the VPR seemed more affected by changes in radiator temperature than by water vapor partial pressure in the drying chamber. Various other factors were expected to influence VPR values such as chamber size, load size, nature of food, and size of pieces so that it was not possible to interpret VPR readings except in largely qualitative terms. An attempt was made to correlate terminal VPR readings with residual moisture content, but no correlation was found even though all runs were made on beef sticks and at the end of all these runs the radiator temperatures were the same.

Figure 6

Comparison of Terminal VPR and Moisture Contents

**R.S.	20	20	20	20	20	20	20	1	1
T. 10	200	350	100	200	600	400	140	100	150
T.M.	1.1	1.9	1.1	1.1	2.3	1.1	1.1	1.0	2.9-1.4
R.S.	1	1	1	1	1	1	1		
T. 10	20	20*	50*	12	4	30	0+		
T.M.	.35-.48	1.1-.97	1.6-1.3	1.3-.99	1.2-.92	1.3-1.3	1.0-.95		
R.S.	1	1	1	1	1	1	1		
T. 10	4	4	20	8	5	5*	0+		
T.M.	2.1-1.2	3.4-1.3	.96-.81	1.5-1.3	1.3-1.1	1.5-1.4	1.1-1.0		

* 20 Sec. VPR
+ read on x1 scale, rest on x.1 or x.01 scales
**R.S. - Run Size (lbs.)
T. 10 - Terminal 10 sec VPR
T.M. - Terminal Moisture (%)

The VPR measurement, unlike imbedded thermocouple measurements, does respond to the condition of the entire charge. The measurement is sensitive enough to be used in following the drying of a single 1/2" x 1/2" x 1" beef stick in a chamber whose volume is over 60 liters. (figure 7) This is not surprising since calculation will show that a rise of 80 microns pressure in this chamber represents the vaporization of just five milligrams of ice.

Figure 7

VPR (microns) of single beef stick during a process cycle.

Time (min.)	30	60	90	140
10 sec. VPR	80	55	15	3
20 sec. VPR	160	100	30	6
40 sec. VPR	330	200	60	11

In spite of the lack of a precise mathematical understanding of the VPR measurement it has still been possible to use it as an empirical end point indicator on a laboratory scale.

In general, during the course of a run the VPR rises from some initial value to either a maximum or a plateau and then decreases eventually to some very low value. (see figures 9,10). On the basis of previous experience it is possible to select some level, below which it is safe to presume that the charge is dry. The VPR technique is especially useful when drying numbers of materials for the purpose of producing samples.

An obvious limitation of the VPR system is that chamber leakage will interfere. Chronic leakage should not be allowed to exist and sporadic leakage would simply indicate drying cycles to be longer than they actually were. The system therefore will "fail-safe". An acceptable leakage rate for a 7' x 7' x 4' chamber would be 5-10 microns/hour. Smaller chambers would have higher values. A less obvious problem is that the sensitivity of VPR readings will be dependent on the operating pressure range since VPR readings must inevitably be taken against the chamber pressure as background. The chamber pressure determines the Alphatron(R) scale being read which, in turn, determines the sensitivity with which a VPR value can be taken.

Vapor Sample Condensing

Because the earlier comparison of Alphatron(R) and McLeod pressure gauges could not be correlated with the progress of the drying process it was decided to see whether trapped and untrapped Alphatrons (R) would give a better measure of the partial pressure of water vapor in the chamber atmosphere. It was found that there was no correlation between the readings of trapped and untrapped Alphatrons and the progress of the process. In some runs the trapped Alphatron read a higher pressure than the untrapped Alphatron. A qualitative explanation of this unexpected reversal is that the pressure at the two sensing heads was equalized since obstructions in the line leading to the trapped Alphatron were kept to a minimum and the trapped Alphatron was exposed to an atmosphere of noncondensable gas and the untrapped Alphatron was exposed to an atmosphere of water vapor. Since water vapor gives a lower apparent pressure reading than air, this could account for this observation except that the size of the reversal was too great to be brought into line by the appropriate correction factor.

VAPOR SAMPLE CONDENSING SYSTEM

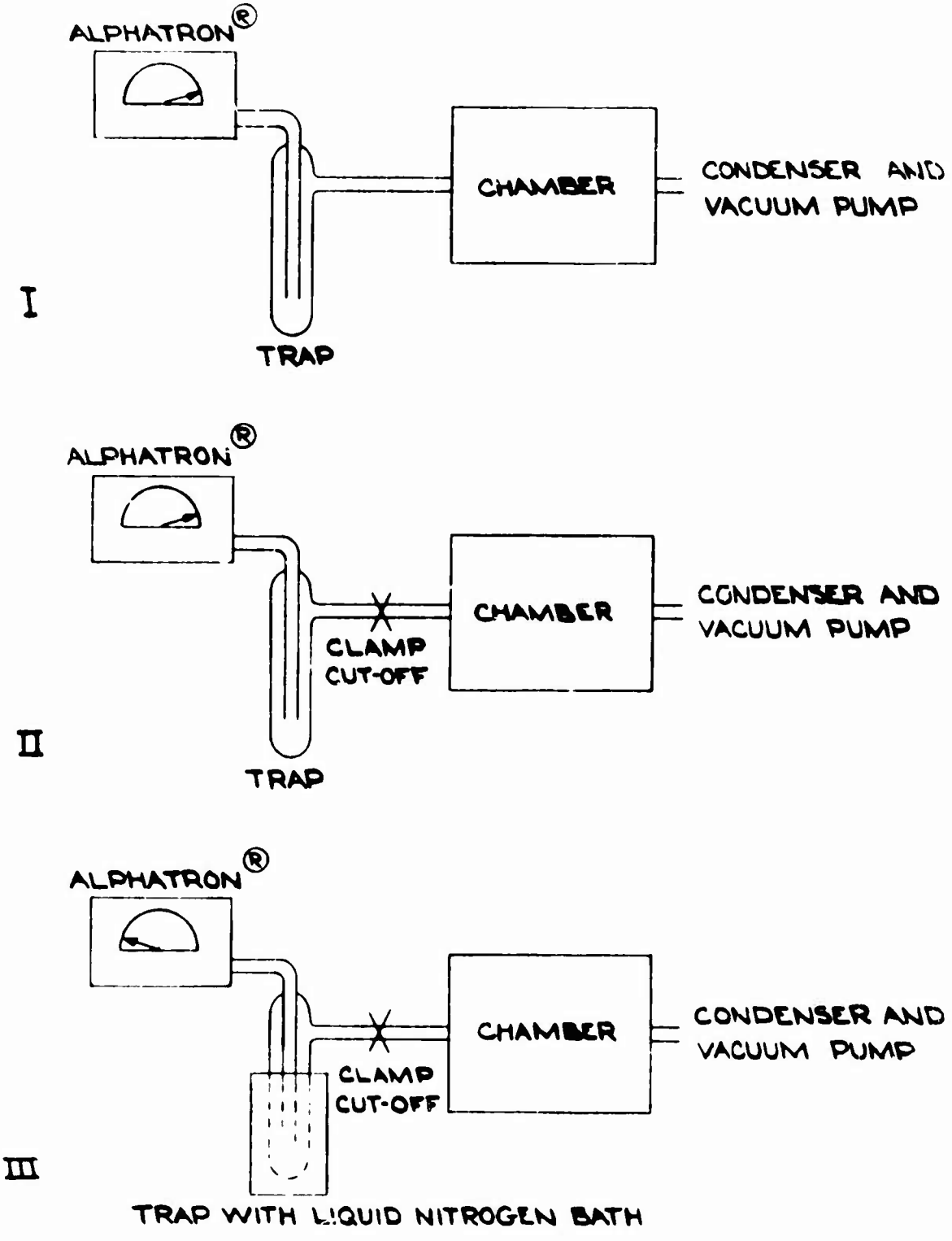


FIG. 8

A-75381

It was decided to operate with just the trapped Alphatron and compare its reading when the trap was uncooled (figure 8-I) with its reading when the trap and Alphatron were isolated from the chamber (figure 8-II) and the trap was cooled with liquid nitrogen (figure 8-III). The results of two runs are shown in (figures 9, and 10).

Figure 9

Vapor Sample Condensing as an end point indicator
(1000 microns operating pressure)

**T.	30	60	90	120	150	180	210	240	300	330
O.P.	900	900	1000	1200	1200	1100	1000	950	1000	1000
C.S.P.	10	10	12	12	20	10	10	11	95	240
VPR	200	500	400	600	500	500	200	100	25	0

Figure 10

Condensing Alphatron^{R} as end point indicator
(100 to 14 microns operating pressure)

**T.	30	60	90	120	150	180	210	240	270	300
O.P.	100	64	46	36	28	22	13	15	14	14
C.S.P.	8	5	3.8	3.2	3.6	3.6	2.8	6	4.4	5.1
VPR	300	300	360	340	360	260	100	20	6	4

**T. - Time (minutes)
O.P. - Operating Pressure
C.S.P. - Cooled Sample Pressure
VPR - 10 Second VPR

In Figure 9 the chamber pressure was maintained at 1000 microns by bleeding air into the refrigerated condenser. In this run the "cooled sample pressure" or CSP remained low throughout most of the run, but rose in the last two readings taken. In Figure 10 the chamber pressure was not controlled and the decrease in total pressure made the end point hard to detect.

The original assumption was that the entire isolated volume comes to equilibrium at the temperature of liquid nitrogen. (-320°F). Calculation of the gas low contraction on this basis predicted pressure drops to values roughly 20% of the original pressures. Tests run on the empty chamber showed that this assumption was false. (figure 11)

Figure 11

Vapor Sample Condensing Values for empty chamber.
Chamber at 200°F. (Pressure control by air bleed)

Initial pressure (microns)	14	54	96	220	500	1000	2000	3200
Pressure after cooling (microns)	5	34	71	200	400	750	1600	2700
Reduced pressure as percent of initial	36	63	74	91	80	75	80	84
Chamber at 70°F								
Initial pressure (microns)	50	170	700	1300	2300	4800	1800	
Pressure after cooling (microns)	14	76	360	880	1800	3800	1400	
Reduced pressure as percent of initial	28	45	51	68	78	79	78	

The VSC system was not rigorously analyzed, but was left as a possible empirical end point indicator that could have advantages over the VPR system in commercial application in that its reading is less sensitive to chamber leakage and does not require the closing of large valves.

It does appear that it will be necessary in commercial practice to raise the chamber pressure to some arbitrary level by bleeding in a non-condensable gas in order to obtain a clear-cut change at the end of the cycle. While this is undesirable, it may not be too serious a drawback particularly if the VSC is applied only towards the end of the drying cycle.

Farvitron as End Point Detector

The Farvitron is manufactured by E. Leybold's Nachfolger. It is a radio-frequency mass spectrometer with rapid response. It measures masses from 2 to 200 and has an oscillographic read-out. Its operating range is from 10^{-4} to 10^{-8} torr, which is below that of the freeze-dryer. It was therefore necessary to assemble the equipment

shown in Figure 12. The oil diffusion pump holds the pressure in the test dome to the 10^{-5} to 10^{-6} torr range even when the chamber is connected to the system. The Farvitron head is thereby exposed to gas molecules flowing from the drying chamber, but at a low enough pressure so that it can be used. This set-up may be thought of as a "pressure transformer".

The results are shown in Figures 13 and 14. Initially (Frame 1) a high peak at mass 28 was observed. This was attributed to nitrogen. As the dehydration began the peak at mass 18 increased to a high value (Frame 2) indicating water vapor and, because of peak interaction in this instrument, the peak at mass 28 was depressed. As the process proceeded to its end (Frames 3 through 8) the mass 18 peak once again decreased and the mass 28 peak correspondingly increased back to its original value. At the end of the run, when the VPR reading indicated adequate dryness the wall temperature was raised to 400°F in an attempt to obtain other peaks due to possible breakdown products. No such peaks appeared. However, the water peak again rose (Frame 9) together with the VPR. After 15 minutes at 400°F the water peak again decreased (Frame 10) and the VPR also dropped to 4 microns. Presumably more tightly bound water was driven out by the drastic heating at 400°F. Two other runs were made with similar results. Thus, it is apparent that the Farvitron can be used to follow the progress of a drying cycle although it cannot be used to detect breakdown products of the beef. If the appropriate end point relationship between H_2O and N_2 peaks is established the Farvitron can be used as an end point indicator. Frame 7 in Figure 10 may be taken as the end point in the example presented. Figure 15 shows the actual pressure values measured during the run.

The Farvitron method of end point determination is related to the VSC system in that a relationship between water vapor partial pressure and nitrogen partial pressure is used to indicate the end of the drying cycle.

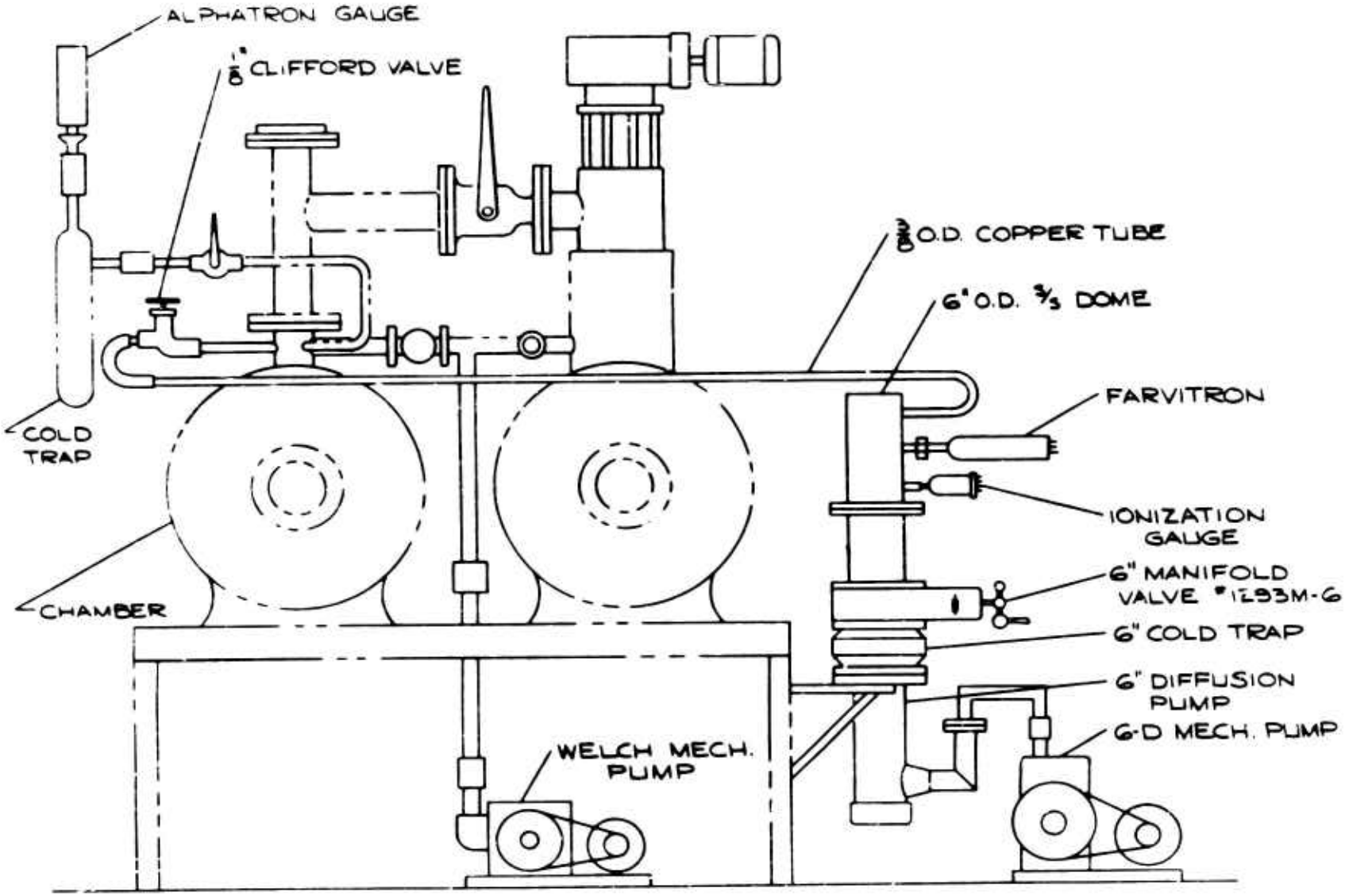
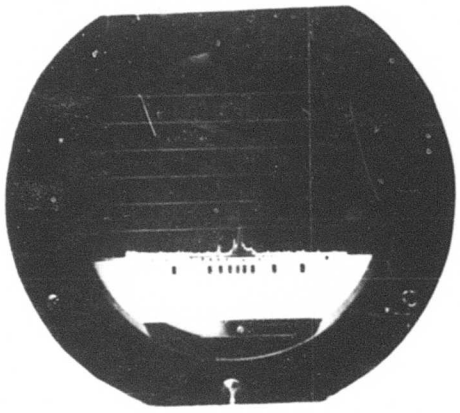
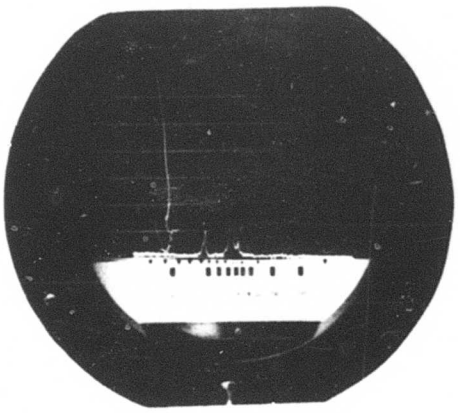


FIG. 12 EQUIPMENT SET-UP FOR FARVITRON TESTS



1



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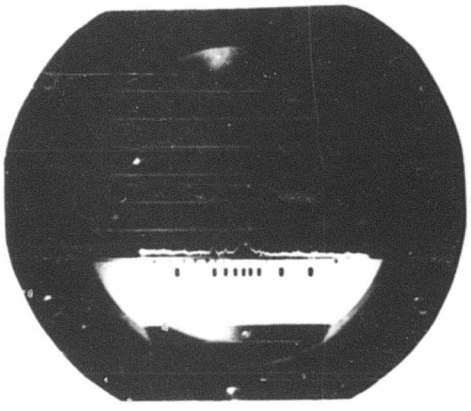

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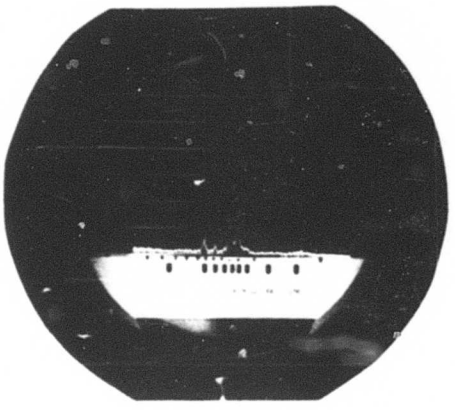

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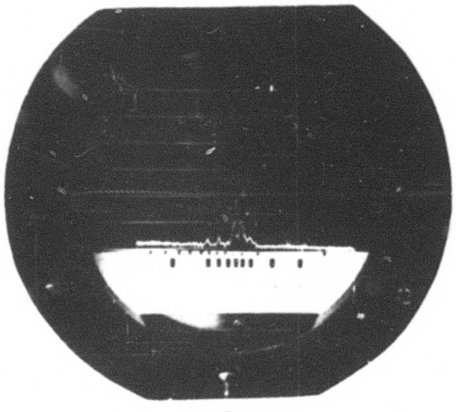
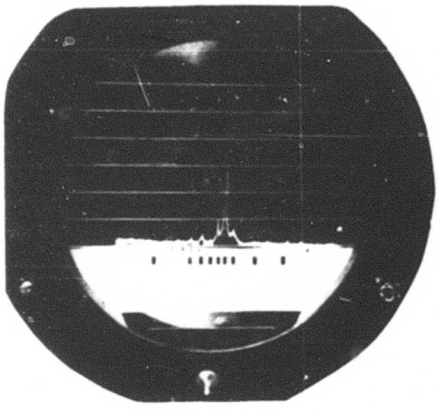


5

Farvitron Read-out



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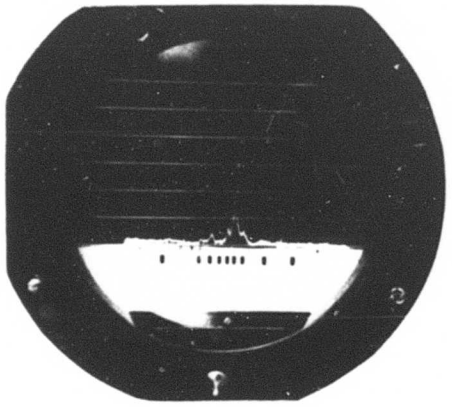

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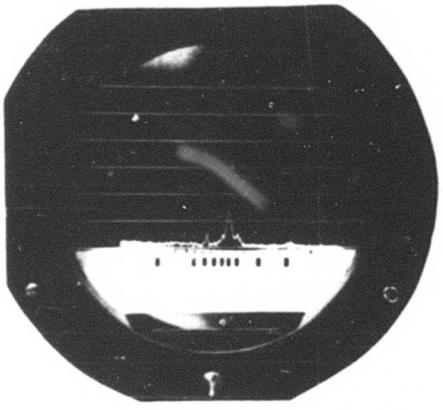

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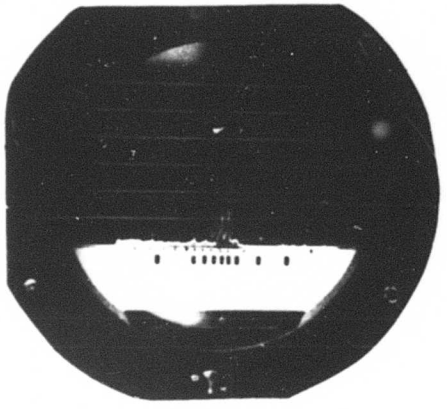

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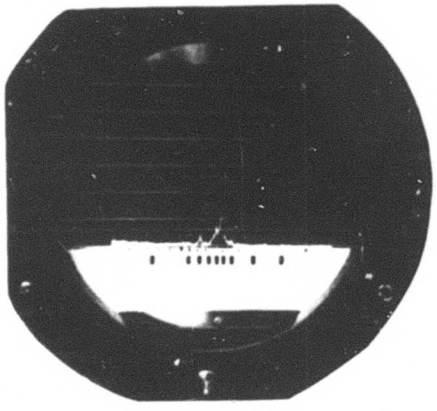


10

Farvitron read-out

Figure 15

Frame	Time hours	Chamber Pressure (microns)	Dome Pressure (x 10^6)	VPR (microns)
1	0	-	2.4	-
2	1	64	5.6	220
3	2	36	3.8	220
4	3	28	3.2	240
5	4	20	2.5	200
6	5	14	2.2	55
7	5 1/2	10	2.0	12
8	6	12	2.0	4
9	6 1/2	26	2.6	80*
10	6 3/4	52	3.8	4

*Chamber wall raised to 400°F.

The difference between the methods is that whereas the VSC requires inleakage sufficient to bring the chamber pressure up to about one torr, the Farvitron registers a background nitrogen peak that is due to inleakage at a much lower pressure. The Farvitron method therefore does not require the opening of a deliberate leak in the system. It has the other advantage of giving a continuous reading.

Infra-Red Detection of Product Surface Temperature

In earlier work with freeze drying of beef sticks it appeared that when operating at total pressures less than one torr it was possible to scorch the meat without causing melting of the ice. High vapor pressure rises indicative of thawing were not observed and the product was in all cases found to have dried without the typical hardening that occurs when beef is dried from the thawed state. It was concluded that product surface temperature was the limiting factor and that it should be sensed and controlled. In addition it was felt that a method of measuring product temperature that was more accurate than thermocouples would be of value as an end point detection method when used instead of thermocouples. There were some hopes that a scanning system could be developed so that temperatures in various places inside a dryer could be read. Attainment of this goal, while

technically feasible, would require considerable additional development. The Barnes Engineering Co., Stamford, Connecticut, manufactures infra-red sensing devices that should be suitable for reading product surface temperatures. Their model R-4D1 Industrial Radiometer was obtained so that the feasibility of this measurement could be established.

Briefly, this equipment consists of two units - a sensing head and an amplifier-power supply. The sensing head contains an optical system of mirrors that focuses target radiation on the radiation detector - a thermistor bolometer. In front of the detector there is a chopper that opens and closes the sensing aperture at a 150 cycle per second rate. The chopper causes the detector alternately to see the incoming radiation and a reference source near the detector. The result is an alternating signal whose peak to peak voltage is proportional to the difference between target and reference source radiation. The amplifier contains a vacuum tube voltmeter, a synchronous rectifier and a temperature monitoring circuit for determining the temperature of the reference black body.

The radiometer head is pointed at the target and is focused either by projecting a focusing light spot or adjusting the distance scale and sighting the target through the periscopic sight. In the experiments reported here readings were then taken on the vacuum tube voltmeter which indicate the output of the radiometer amplifier.

The first experiments were an attempt to follow the surface temperature of beef as it was being freeze dried. The radiometer was focused through an infra red transparent germanium sight port onto a piece of beef inside the drying chamber. The beef also had a thermocouple imbedded in its surface.

It was found that a qualitative relationship between radiometer reading and thermocouple reading could be established. In other words, the radiometer did indicate the initial temperature, a decrease in temperature as the chamber was evacuated followed thereafter by a steady temperature rise. Two difficulties were encountered: 1) the germanium window attenuates the signal and this attenuation was apparently different at the beginning and end of the run, and 2) the

equipment has a noise zone around ambient temperature wherein no meaningful readings can be obtained and the noise zone is effectively enlarged by the attenuation due to the germanium window.

In the second group of experiments various freeze dried foods were fitted with thermocouples on their surfaces and placed in the chamber at atmospheric pressure. The germanium window was removed and the surface temperature of the food was read directly through the aperture. The temperature of the food was raised by the heated chamber walls. The readings obtained are shown in Figures 16 through 20. It is apparent that the emissivity of the various foods tested must be quite similar and all of them seem to be high which is favorable, since the likelihood of reflectance of energy from the hot radiators is reduced. It is not possible to determine which values are absolutely correct since the radiometer is considered the unknown and the thermocouples themselves are subject to error.

In application, however, it would not be necessary to establish an absolute value for the temperature at which scorching occured so long as this value could be reproducibly measured by the radiometer.

Use of a germanium sight port appears impractical because it is not possible to see through it to focus. In addition, the problem with variable signal attenuation was not solved. It may be possible to operate the radiometer head inside the vacuum chamber. If this were the case the radiometer could be used without modification to monitor a belt drying process, but a system to permit scanning of the various levels would still have to be developed to enable use of the radiometer to monitor a cabinet shelf dryer.

FIG 16

CORRELATION : RADIOMETER VS. THERMOCOUPLE TEMPERATURE READINGS

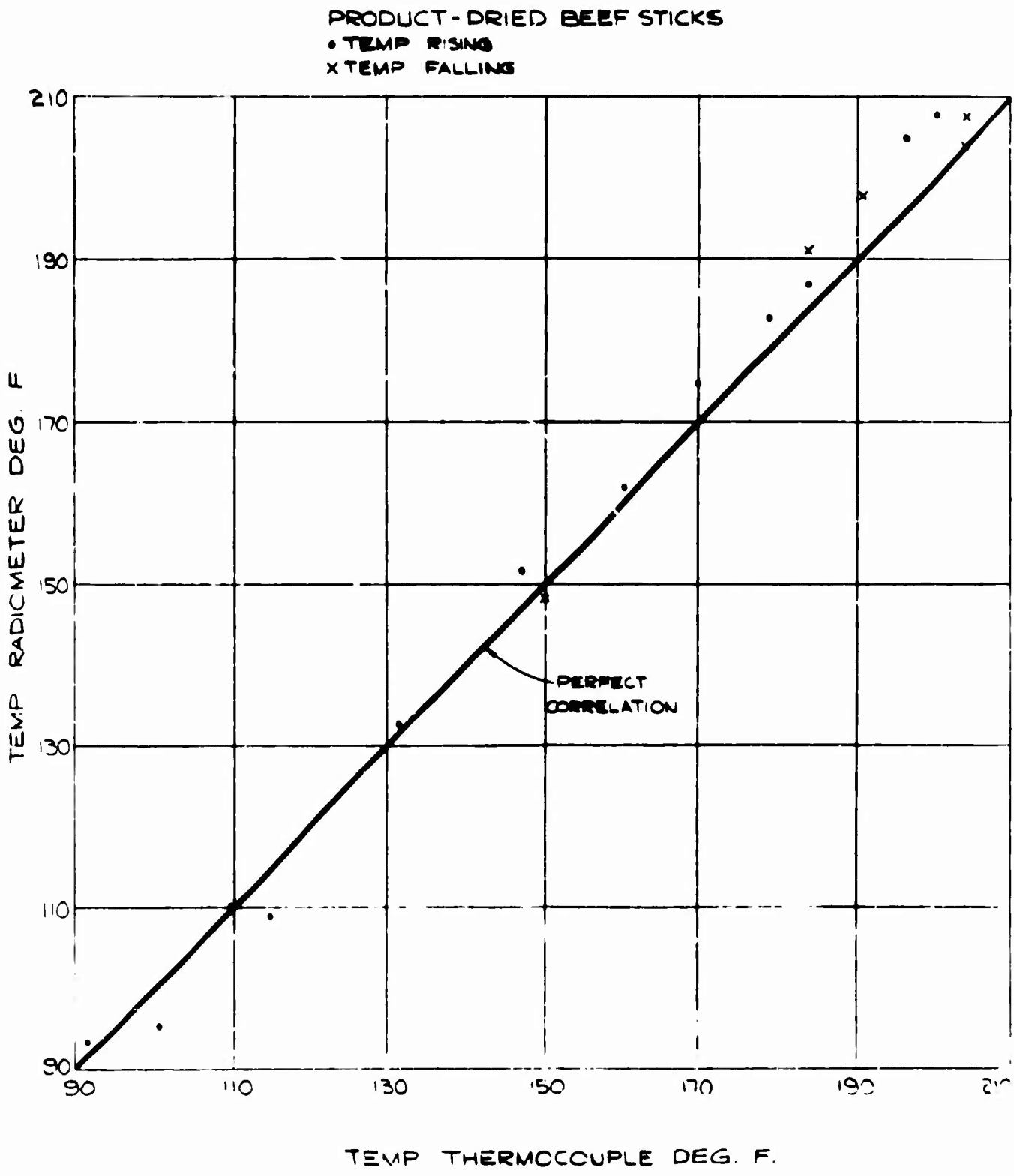


FIG 17

CORRELATION : RADIOMETER VS THERMOCOUPLE TEMPERATURE READINGS

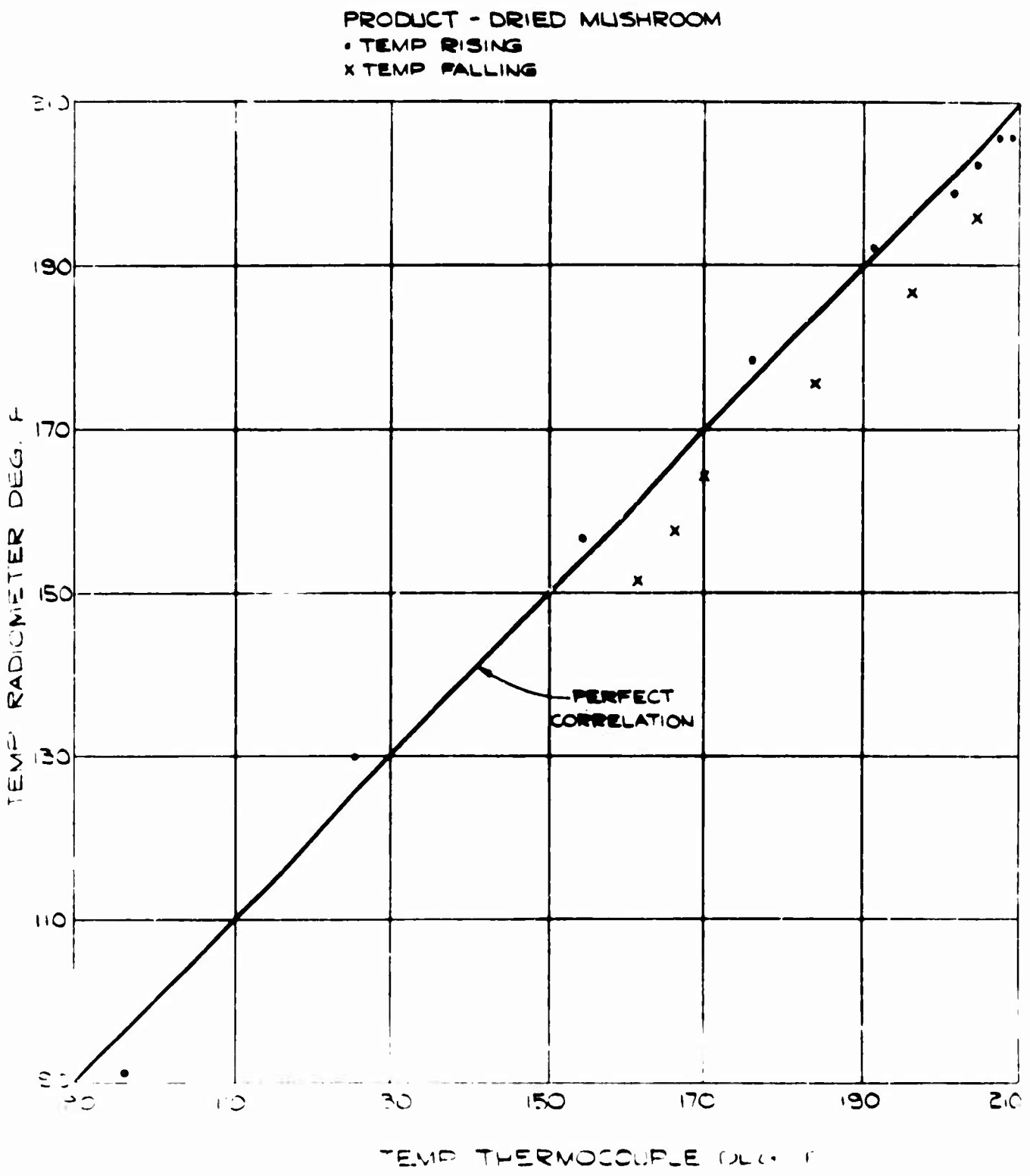


FIG 18

CORRELATION: RADIOMETER VS THERMOCOUPLE
TEMERATURE READINGS

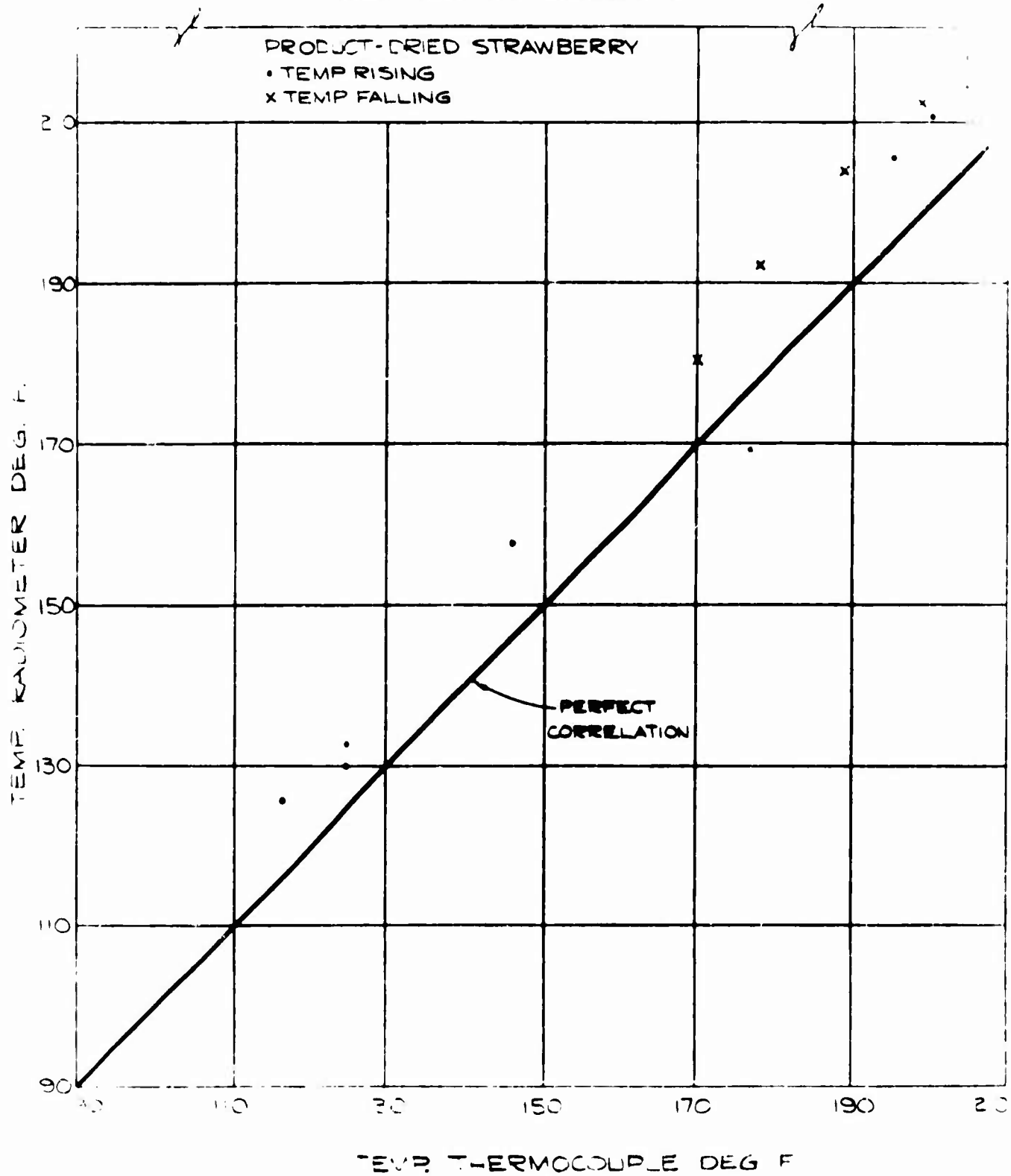


FIG 19

CORRELATION : RADIOMETER VS THERMOCOUPLE TEMPERATURE READINGS

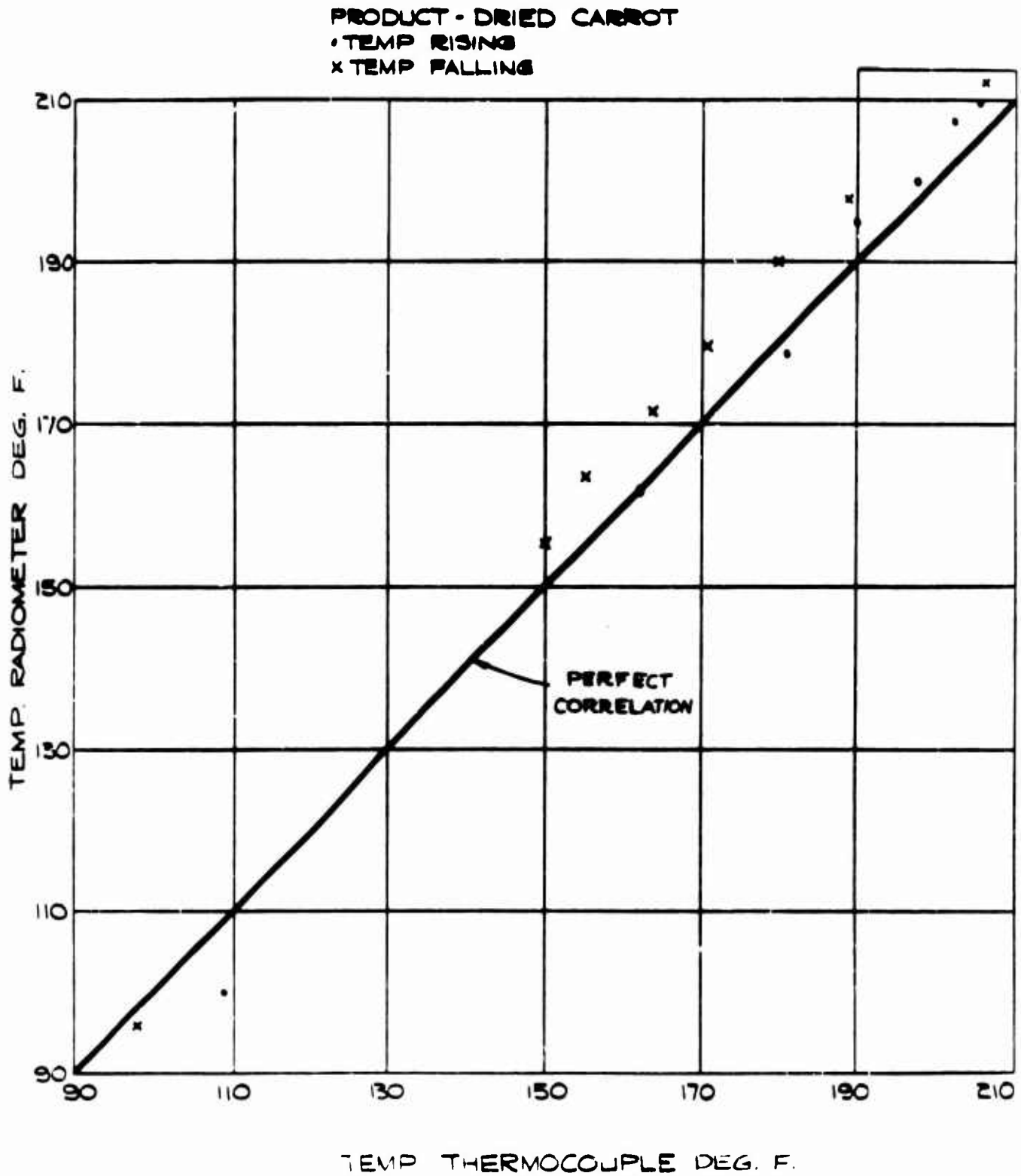
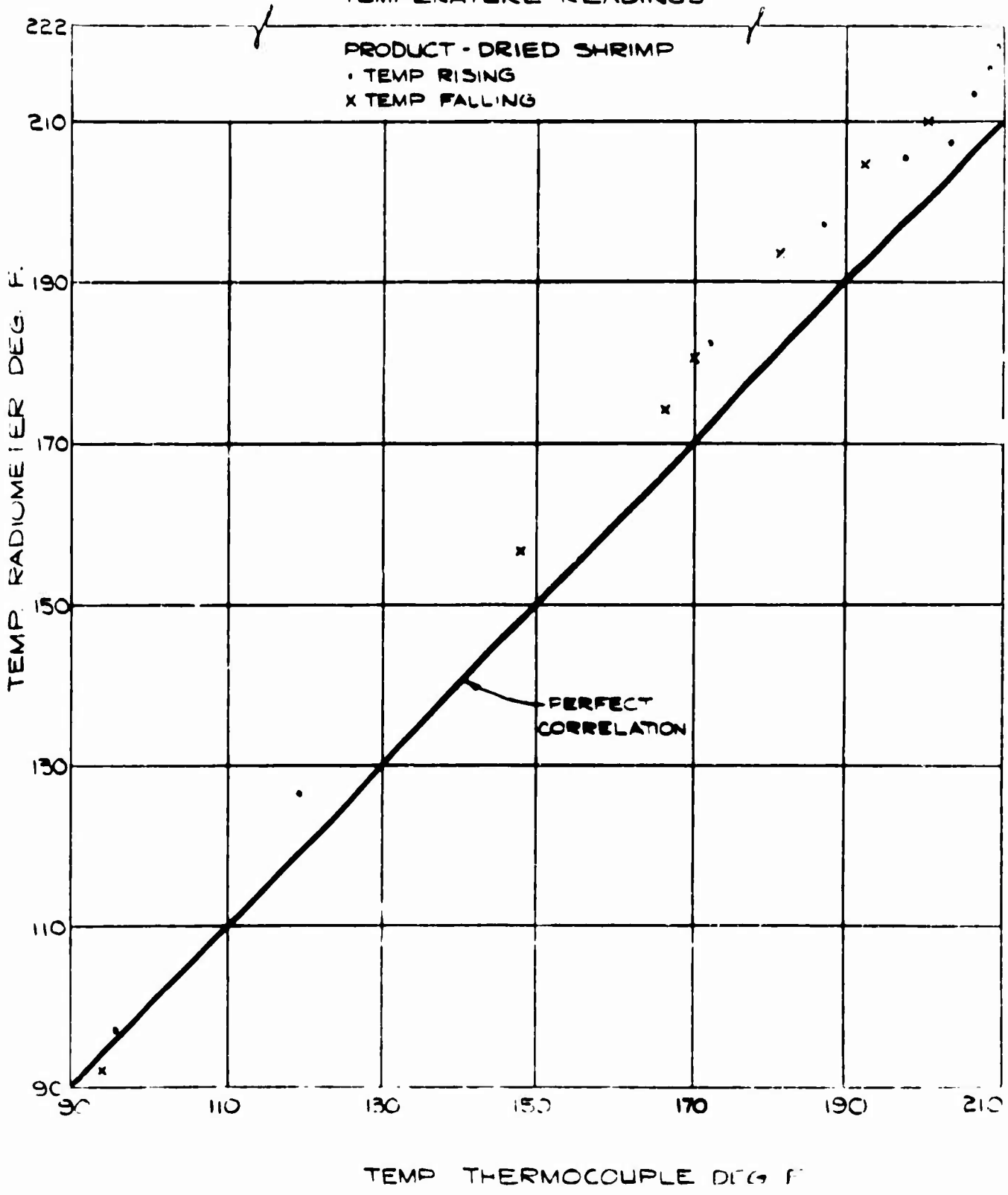


FIG. 20

CORRELATION: RADIOMETER VS THERMOCOUPLE TEMPERATURE READINGS



In-Process Moisture Analysis

A. General

As mentioned earlier, the measurement of the moisture content of the entire load of food as it dries does not have much promise as a method of process end-point detection if a sharp indication is required. It is conceivable that a method with sufficient sensitivity could be used to follow a drying process to some empirically established end point value.

A variety of possible methods of detecting moisture in food were examined including dielectric, nuclear and ultrasonic. None of the methods first examined appeared applicable to the control of freeze dehydration processes. At this point the services of Lion Research Corporation were retained as experts in biological instrumentation and the primary recommendation of their report (reproduced in progress report number 5) was that RF conductivity or power absorption measurements showed promise of correlating with moisture content. Concurrently it was learned that an instrument of this type was available commercially for moisture measurement from the Boonton Polytechnic Company, Rockaway, New Jersey. Earlier work with a dielectric moisture analyser had not produced usable results because it measured the capacitance of the sample which, in turn, was influenced by the samples fat content (quite variable in the case of beef sticks). The Boonton instrument was not expected to be affected in this way.

The Boonton Polytechnic Company developed a special electrode EF-8 for use in this investigation. It consisted of two blackened, perforated sheets of 3/16 inch thick aluminum that were held in aluminum frames. The entire assembly was made so that it would fit between the electrically heated radiators in the NRC freeze dehydration unit. Suitable electrical connections were made with RF cables between the electrode and an Amphenol feed-through installed in the chamber door and from there to the instrument read-out.

The read out was the standard Boonton Polytechnic Company HFR-4E modified to operate at 50 volts RF output. This increase in voltage was necessary because the EF-8 electrode was designed to be non-contacting.

In operation, the EF-8 electrode was used as the product carrier. After nulling the electrode to a minimum reading of 3, one pound of frozen food was placed in the electrode and the two parts of the electrode were fastened together mechanically in order to minimize vibration of the assembly. The food rested on the lower electrode and did not touch the upper electrode. The assembly was placed between the electrically heated platens spaced six inches apart and freeze drying was carried out in the usual manner.

B. Drying of Beef Sticks - Calibration Curve

In the first run it was established that the electrode could be used in a vacuum and the results of a typical run are shown in Figure 21. It is apparent that the moisture analysis could be used to follow the loss of ice as the drying proceeded. The Boonton analyser readings are in arbitrary units. In normal use the instrument must be calibrated against standard samples of known moisture content.

The initial reading on one pound of frozen beef sticks, taken before the chamber was evacuated, was 300 in the run recorded in Figure 21. As the chamber was evacuated to a pressure below 4 torr the analyser reading was observed to rise to a considerably higher value, reaching a maximum in about 8 minutes. This maximum occured when operating pressure had been reached and it was found possible to affect the analyser reading by isolating the chamber and allowing the chamber pressure to rise. An inverse relationship appeared to exist between chamber pressure and analyser value. Since subsequent studies showed that the high reading after the pull-down corresponded roughly to the expected initial weight of moisture in the charge it was decided that the low initial reading would be disregarded and, since this starting phenomenon appeared not to affect the utility of the instrument in monitoring the drying process, no concerted effort was made to explain this unexpected behavior.

A series of runs were made with one pound loads of beef sticks in which drying was stopped at various Boonton analyser readings short of dryness. At the termination of each run the charge was weighed and then completely dried in order to obtain a measure of the weight of

Figure 21

Typical results with Boonton analyser EF-8 electrode using 1 pound of beef sticks 2/21/62

Time	Total Pressure (u)	Boonton Reading	Radiator Temperature (°F)	Vapor Pressure Rise 10 sec. (u)
8:30	Start	300	-	-
9:00	62	1280	250	60
9:30	52	1160	200	70
10:00	46	980	200	90
10:30	46	850	200	120
11:00	43	740	200	150
11:30	40	625	200	180
12:00	38	520	200	190
1:00	32	310	200	240
1:30	28	220	200	260
2:00	25	154	200	240
2:30	22	95	200	210
3:00	16	49	200	140
3:30	12	30	200	50

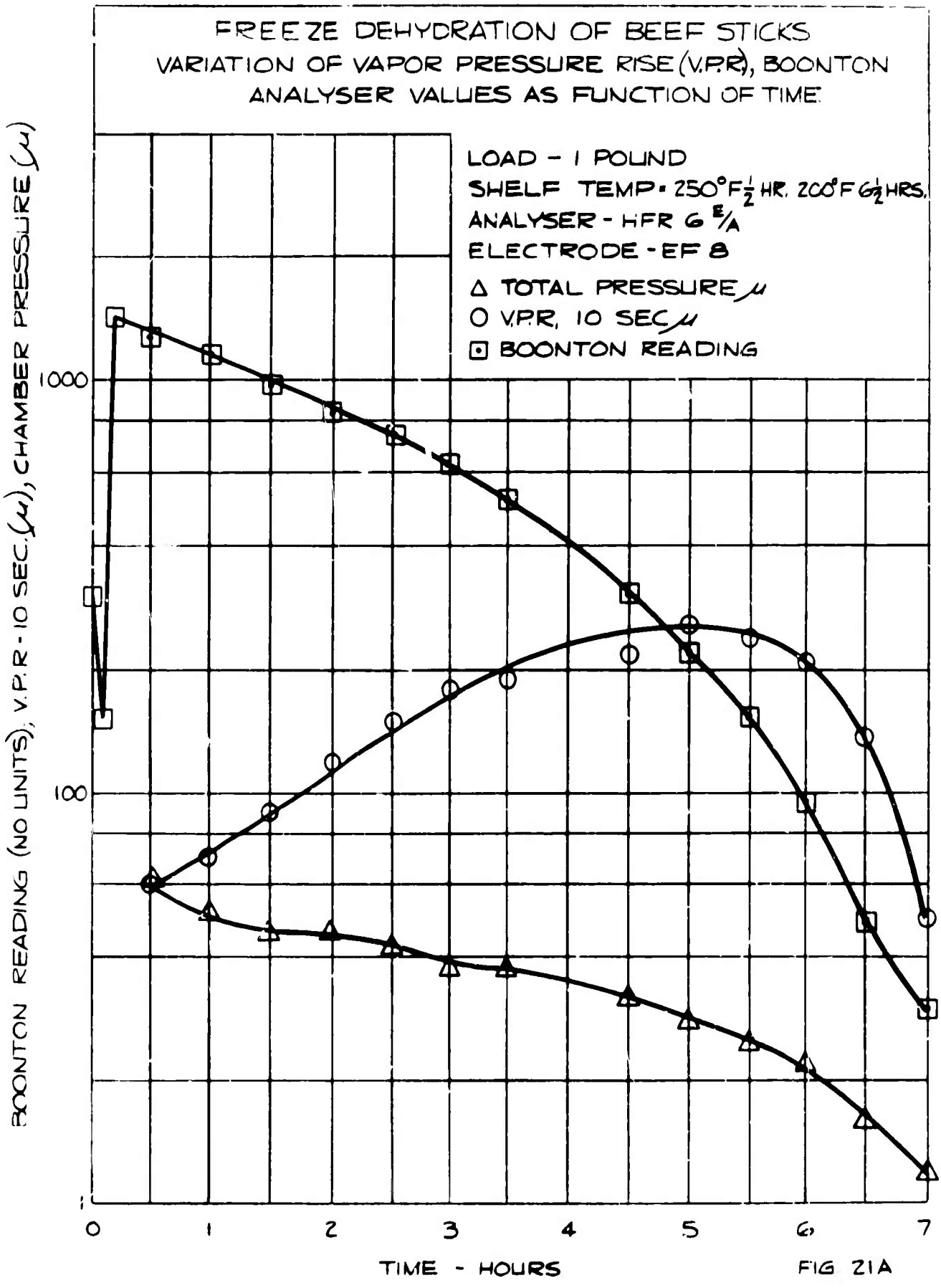

FREEZE DEHYDRATION OF BEEF STICKS
VARIATION OF VAPOR PRESSURE RISE (V.P.R), BOONTON ANALYSER VALUES AS FUNCTION OF TIME
LOAD - 1 POUND
SHELF TEMP - 250°F ½ HR. 200°F 6½ HRS.
ANALYSER - HFR 6 E/A
ELECTRODE - EF 8
Δ TOTAL PRESSURE μ
O V.P.R. 10 SEC μ
⊡ BOONTON READING
BOONTON READING (NO UNITS), V.P.R - 10 SEC. (μ), CHAMBER PRESSURE (μ)
1000
100
1
0
1
2
3
4
5
6
7
TIME - HOURS

FIG 21A

moisture corresponding to the final analyser reading taken in each run. The results are shown in Figure 22 as a plot of analyser readings against residual moisture.

The relationship of analyser values to residual moisture in beef sticks fall on a curve that is close to a straight line. As indicated in Figure 22 stopping runs at three different temperatures did not affect the reliability of the instrument. Since dielectric loss is affected by temperature (see our progress report number 6) it was necessary to determine if radiator temperature would influence the analyser reading.

Four runs were made using beef-sticks that had been boiled for an hour and then refrozen. Three of these points deviate a little from the raw beef curve. This may be attributed to geometric factors since the cooked beef sticks were not as uniform in size and shape as the raw beef sticks which were sawed to size while frozen.

It should also be noted that the curve does not go to the origin since even beef sticks dried to the point where no further weight loss can be found by vacuum oven drying to produce a Boonton reading of about 10 units.

C. Drying of Corn

Commercially available loose frozen whole kernel corn was used in a series of runs in order to establish calibration curves for this product. The moisture content of the corn at various analyser readings was again determined by vacuum drying overnight at 100°F, 125°F and 150°F. The temperature at which the moisture analysis was made appeared to affect the results, but this phenomenon could not be explained.

A fourth series of runs was made in which the corn was comminuted in a Waring blendor and then frozen as a rectangular block before being dried in the usual manner. This was done in order to produce a more uniform sample. This fourth series of runs produced a smooth curve whose slope differed from the curve obtained when the whole kernel corn was re-dried at 150°F. The difference in slope can be attributed to

FIGURE 22

RELATIONSHIP BETWEEN BOONTON ANALYSER VALUES AND RESIDUAL MOISTURE - BEEF STICKS (1 LB. NET)

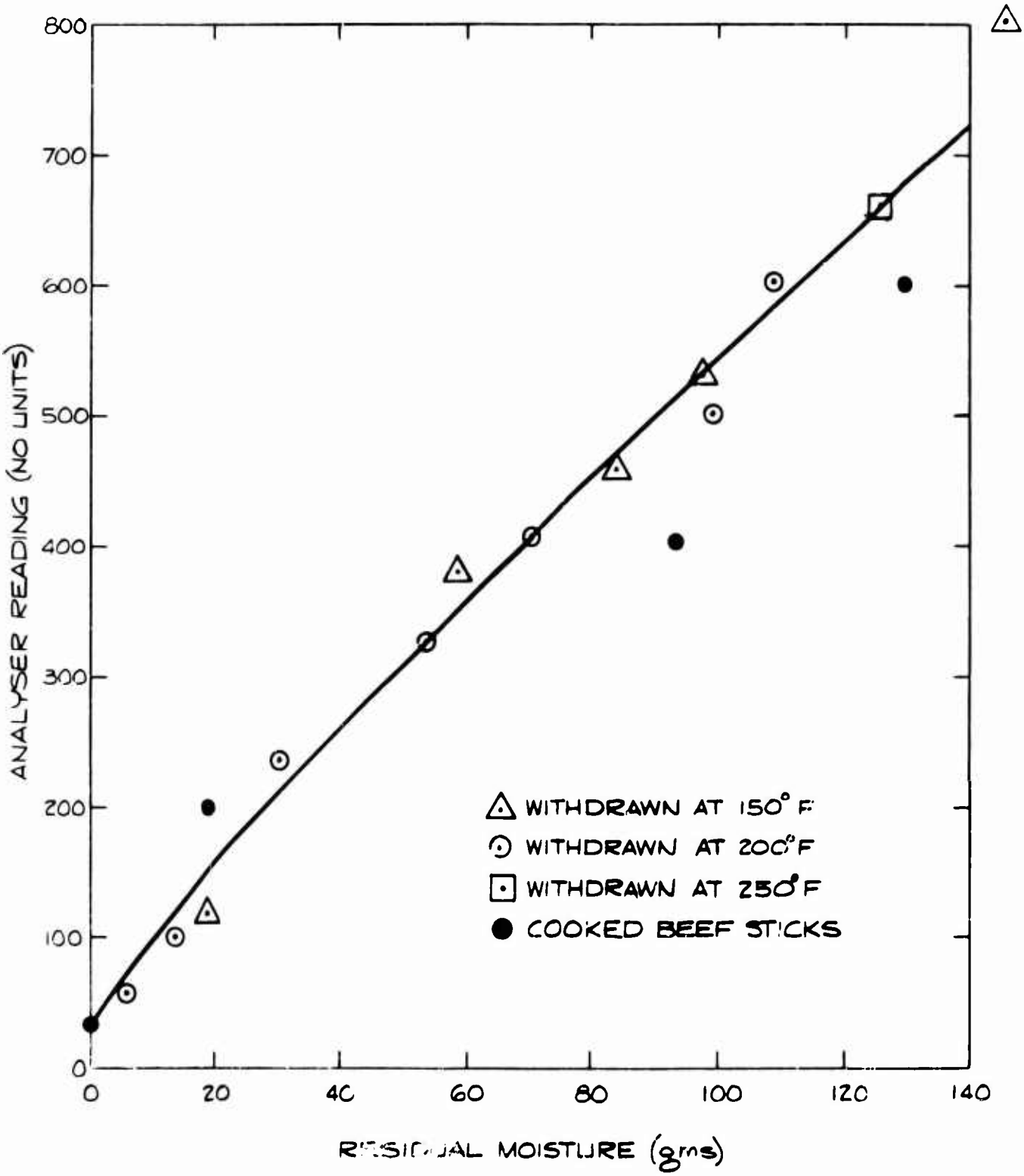
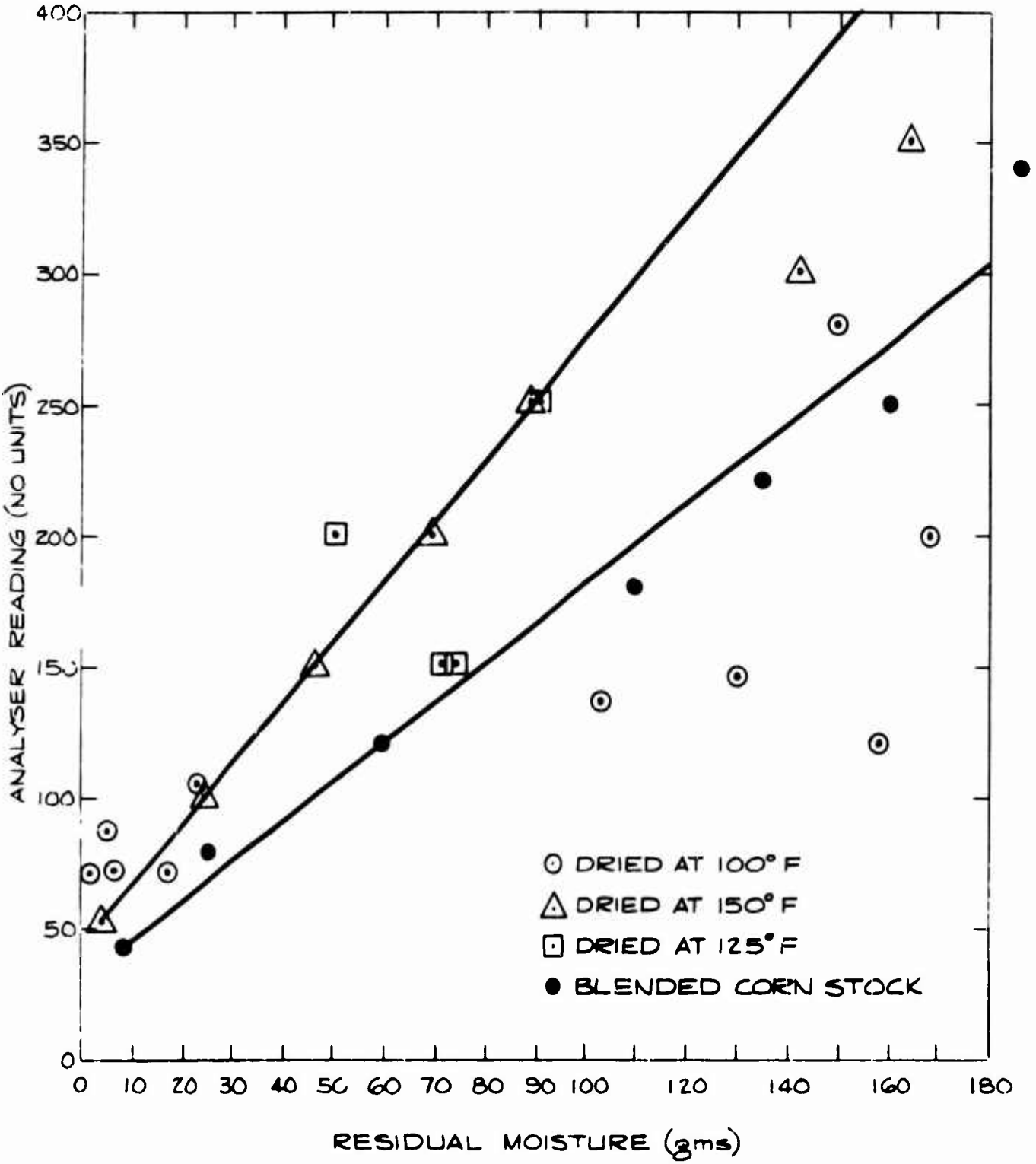


FIGURE 23

RELATIONSHIP BETWEEN BOONTON ANALYSER VALUES AND RESIDUAL MOISTURE - WHOLE KERNEL CORN (1 LB. NET)



the difference in product geometry, but the smoothness of the curve suggested that no significant variations in product composition existed between runs in the set.

D. Other Calibration Curves

In order to evaluate the Boonton analyser over a wider range of products calibration curves were made for loose frozen cut green beans, pineapple chunks and chopped onion. All were procured in the market and none was especially selected. All runs on each product was made with the same brand of material. The results are presented in Figures 24,25, and 26. The curves for all three products appear to be smooth and no single point deviates far from the general trend. The slopes of the three curves are different, as expected. The curve for the pineapple chunks is the first one to appear concave with response to mass of water increasing at the high end. All the pineapple runs were made with radiators at 100°F because at higher temperatures there was excessive product shrinkage during drying.

E. Comments

It is apparent from these experiments that it is possible to follow the progress of freeze dehydration of several foods using the Boonton moisture analyser and that its sensitivity at low moisture contents is sufficiently high to permit its use as an empirical endpoint indicator for the entire load. As was known before, the instrument reading is affected by sample geometry and composition so that in practice it will be necessary to assess the magnitude of these effects in any given application.

One method of applying this procedure to the monitoring of commercial batch drying would be to equip each product level with electrodes, preferably by modifying existing hardware electrically to serve as the electrodes. This would permit monitoring of every piece of food in the entire chamber, but would read only the total moisture content of each product level. A refinement of this procedure would result from segmenting the large electrodes to permit the taking of readings over smaller areas of food or devising an electrode that could be moved about to take readings in different places.

FIGURE 24

RELATIONSHIP BETWEEN BOONTON ANALYSER VALUES AND RESIDUAL MOISTURE - CUT GREEN BEANS (1 LB. NET)

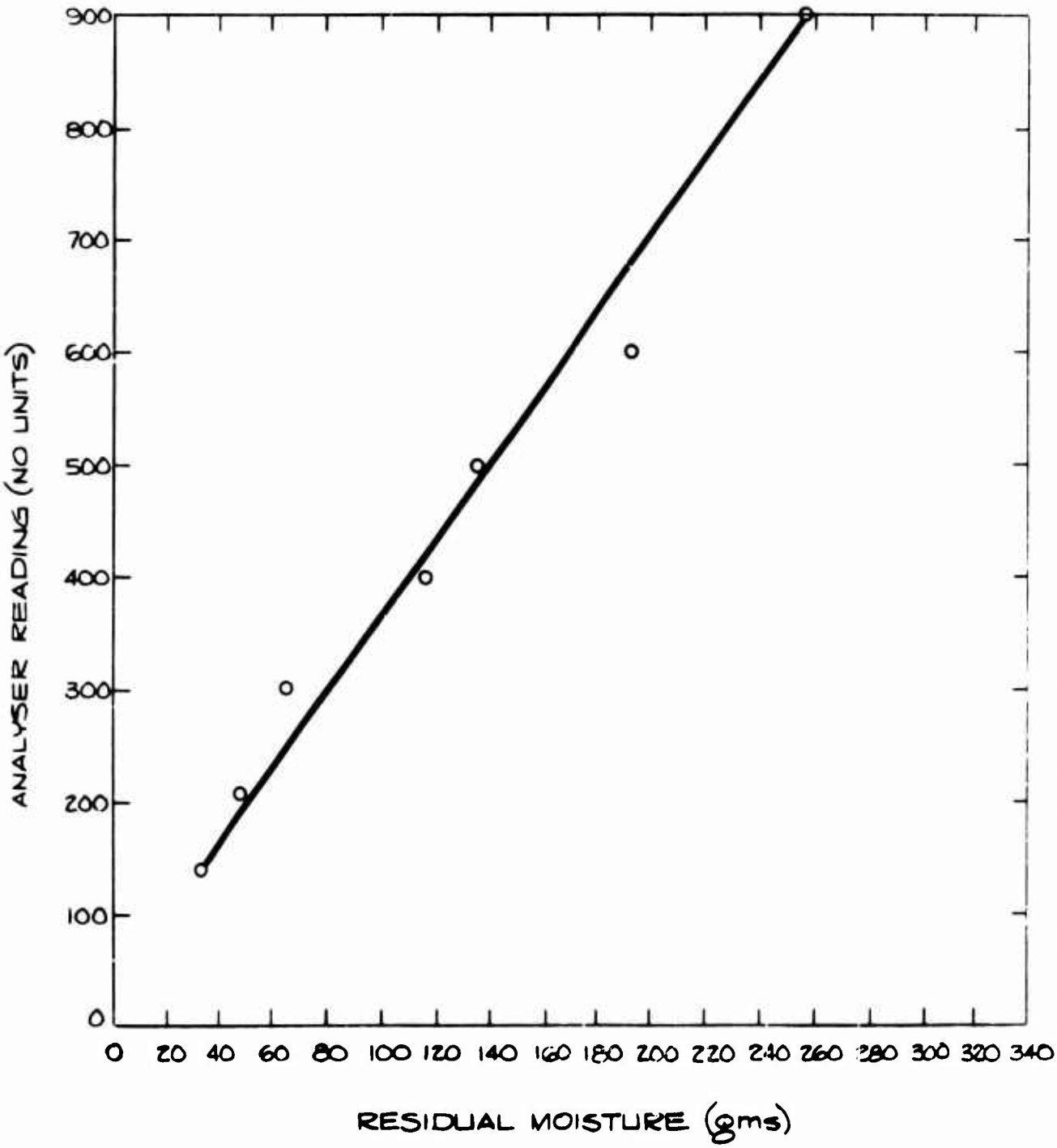


FIGURE 25

RELATIONSHIP BETWEEN BOONTON ANALYSER VALUES AND RESIDUAL MOISTURE - PINEAPPLE CHUNKS (1 LB. NET)

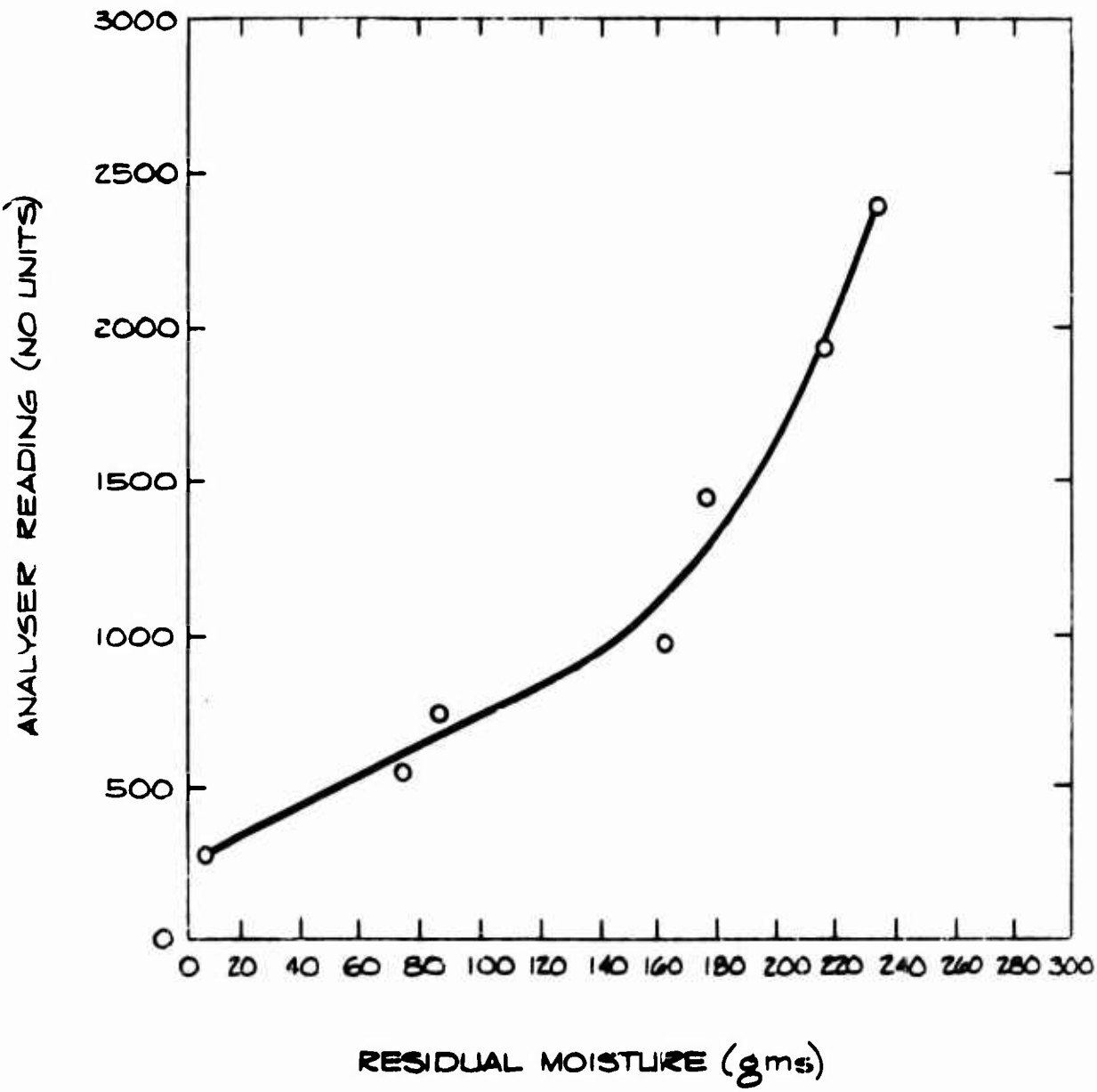
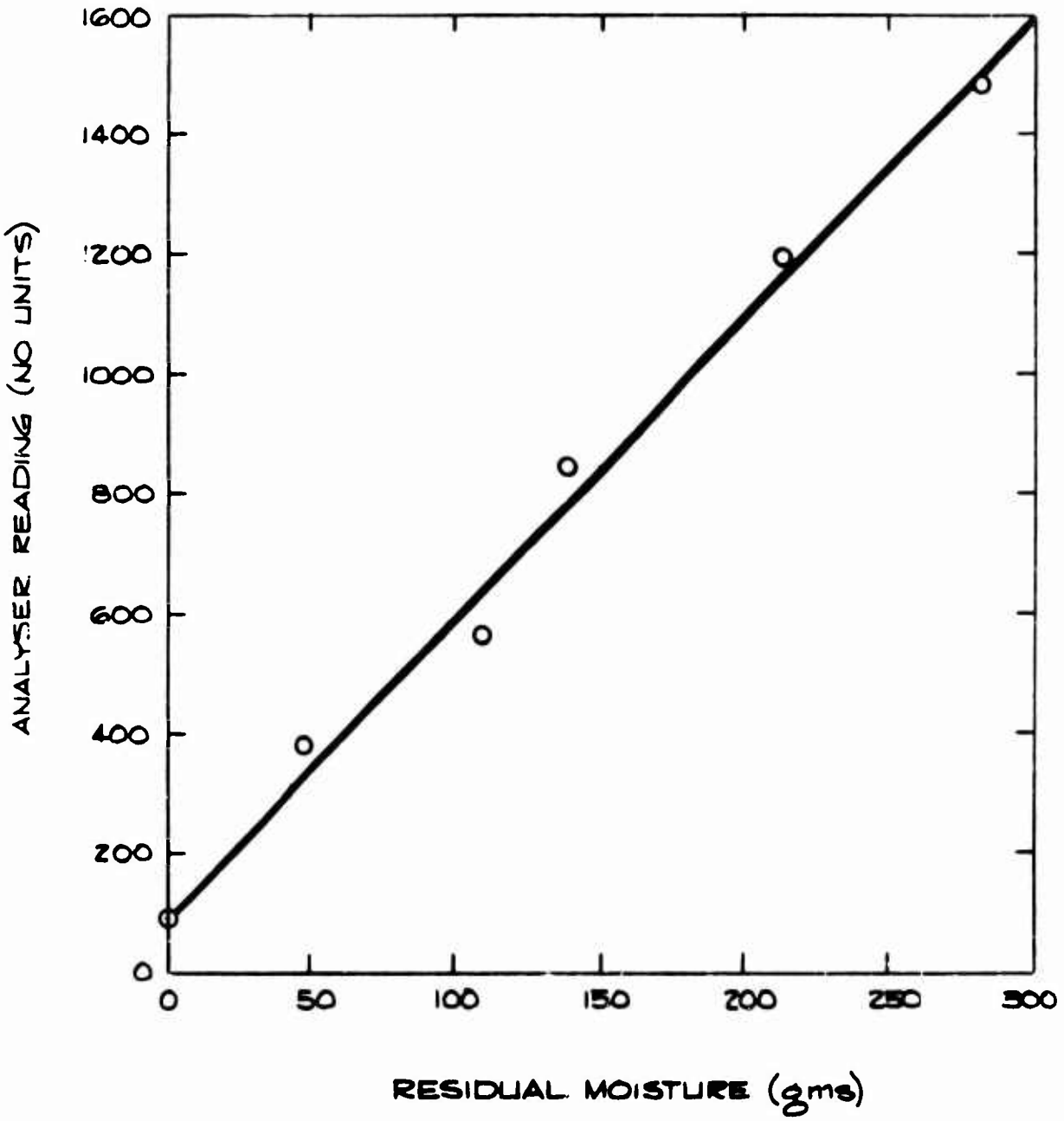


FIGURE 26

RELATIONSHIP BETWEEN BOONTON ANALYSER AND RESIDUAL MOISTURE - DICED ONIONS (1 LB. NET)



Overall Conclusions

During the course of this project a number of approaches have been examined as possible means of monitoring the course of freeze dehydration processes. The VPR, VSC, Farvitron, infra-red radiometer and RF moisture analyser have been examined in depth and all with the exception of the radiometer have been applied successfully on a laboratory scale as monitors of the drying process. It is likely that the radiometer could also have been applied had the necessary equipment modifications been made. All of the five most promising techniques appear applicable on a commercial scale and all of them merit further development effort in that direction Throughout this project an attempt has been made to seek an end-point indicator that would give a sharp signal of some sort that would indicate the process was at an end and this signal was to be obtained by some measurement over the entire chamber load. No such end point sensor has been found; perhaps because no sharp end point exists. Towards the end of the project it has become apparent that one way of obtaining more accurate control of the process is to make multiple measurements over various small areas of the load. This could be achieved, after the exercise of considerable mechanical and electrical ingenuity, in present systems; or, with relative ease, after the exercise of comparable diligence in the development of a continuous conveyorized system.

For the control of present day units it would seem wise to spend at least some effort in trying to apply the three integrating systems of VPR, VSC or Farvitron before fully developing the infra-red radiometer or RF moisture analyser methods. Only infra-red radiometry and RF moisture analysis appear practical for monitoring a conveyorized continuous system.

Appendix

Suppliers of instrumentation contacted during the course of this project.

Moisture Analysers

Forte Engineering Co., Norwood, Massachusetts
Boonton Polytechnic Co.,Rockaway, New Jersey
Moisture Register Co., Alhambra, California
Consolidated Electrodynamics Corp., Pasadena, California

Radiometers

Barnes Engineering Co., Stamford, Connecticut
Servo Corporation of America, Hicksville, New York
Williamson Development Co., West Concord, Massachusetts
Block Associates, Inc., Cambridge, Massachusetts
Epic Inc., New York, New York

Nuclear Measurements

Nuclear-Chicago, Des Plaines, Illinois
Industrial Nucleonics Corp., Columbus, Ohio
Nuclear Enterprises Ltd., Edinburgh, Scotland

Miscellaneous

R. I. G., Schlumberger,/Inc, Ridgefield, Connecticut
General Mills, Mechanical Division, Framingham, Massachusetts
Minneapolis-Honneywell Co., Minneapolis, Minnesota
Cambridge Systems, Inc., Waltham, Massachusetts
Beckman Instruments, Fullerton, California
Foxboro Co., Foxboro, Massachusetts

Progress Report Contents

Number 1

Alphatron[R] - McLeod gauge correlation.

Initial studies on vapor pressure rise. (VPR).

Number 2

VPR correlation with ice temperature.

VPR correlation with residual moisture content.

VPR as end point indicator.

Effects of wall temperature and chamber pressure on drying curves.

Moisture content of beef sticks at point where last ice is just being sublimed.

Vapor sample condensing (VSC) investigated as end point indicator.

Number 3

VSC limitations discussed.

Infra-red radiometer investigated for process control.

Number 4

Farvitron evaluated for end point determination.

Vapor transfer study - effect of drilled holes on drying of beef sticks.

Number 5

Vapor transfer study concluded.

Sorting systems suggested.

X-ray transmission evaluated as a sorting measurement.

Dielectric conductance measurement recommended for moisture analysis.

Number 6

Dielectrometer values for frozen and freeze dried beef.

Non-uniformity of drying within a load of food demonstrated.

Number 7

R F moisture analyser evaluated for process control.

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